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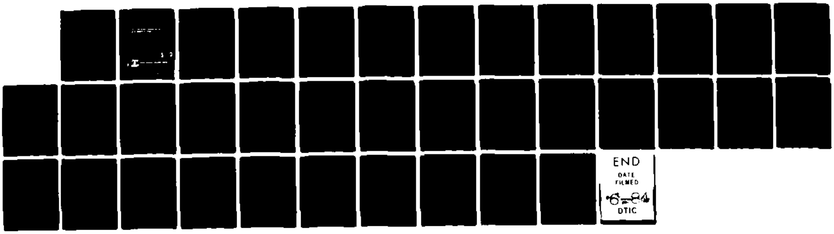

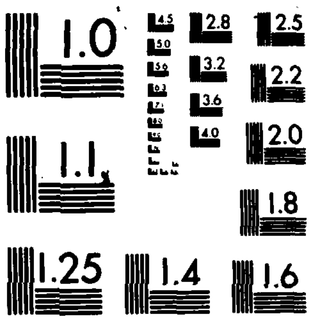

MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

AD-A140 650

NSWC TR 83-416

EVALUATION OF MICA SUBSTITUTES FOR USE IN THERMAL BATTERIES

BY S. DALLEK, B. F. LARRICK (NSWC)
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RESEARCH AND TECHNOLOGY DEPARTMENT

1 SEPTEMBER 1983

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FOREWORD

This work was undertaken in response to a general need within the thermal battery industry to find a substitute for the critical and strategic material phlogopite mica used as high temperature electrical insulation in thermal batteries. Possible substitute materials were studied by actual configurational tests in batteries and by thermogravimetry (TG).

The support of the Design Options for Substitute Materials Program of NAVSEA is gratefully acknowledged.

Approved by:

J. R. Dixon

JACK R. DIXON, Head
Materials Division

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CHAPTER 1

INTRODUCTION

Phlogopite mica is a complex naturally-occurring hydrous aluminum silicate mineral that is used as electrical insulation in thermal batteries. The idealized formula for phlogopite is $K_2Mg_6Al_2Si_6O_{20}(OH)_4$ but several other elements may be incorporated into the structure as isotopic replacements.[1] The United States is almost completely dependent on foreign sources for strategic sheet mica.[2] The Government has thus classified it as a critical and strategic material and must therefore maintain a stockpile for use in emergencies.

The objective of the present work was to perform an experimental evaluation to identify and support the development of a substitute material for phlogopite mica in thermal batteries. Candidate materials must be thin (0.003-0.010 in) and flexible to wrap around the cell stack and must maintain good electrical resistance and thermal stability at thermal battery operating temperatures (500°-550°C). Chemically, the material must be resistant to molten salts, strong oxidizing and reducing agents, and should be non-hygroscopic.

Candidate materials were studied by thermogravimetry (TG) to determine the decomposition temperature and mass loss of each sample. The materials were also subjected to actual configurational tests in thermal batteries employing the $LiAl/LiCl\text{-}KCl,SiO_2/FeS_2$ molten salt electrochemical system.

[1]Skow, M. L., U. S. Bureau of Mines Information Circular 8125, "Mica, A Materials Survey," (1962).

[2]Petkof, B., U. S. Bureau of Mines Bulletin 630, "Mica" in Mineral Facts and Problems, 1965 edition, p. 583-94.

CHAPTER 2

EXPERIMENTAL

THERMOGRAVIMETRY (TG)

A DuPont 1090 Thermal Analysis System with a 951 Thermogravimetric Analyzer was employed in this study. In preliminary tests to study the decomposition behavior of phlogopite mica and various candidate replacement materials, samples were run in platinum boats at a heating rate of 20°C/min under a flowing atmosphere of dry argon to a maximum temperature of 1150°C. In all subsequent runs, the TG heating program was modified to simulate the actual temperature vs. time profile experienced by internal thermal battery components, i.e., a maximum average temperature of 500°C-550°C for periods up to 30 minutes. The sample was either heated at 100°C/min to about 500°C and then isothermally for about 30 minutes or was inserted into a preheated furnace at 500°C for 30 minutes. The candidate replacement materials for phlogopite mica evaluated in this study are listed in Table 1.

BATTERY TESTS

Batteries were constructed and discharged to compare the electrical properties of the various candidate insulating materials. The electrical noise level and life of the battery were used to evaluate the thermal stability and electrical insulating capability of the materials. In addition, post mortem analyses were performed to assess the degradation of the insulators. The details of construction of a typical battery are shown in Figure 1.

CHAPTER 3

RESULTS AND DISCUSSION

A TG curve of phlogopite mica is shown in Figure 2. The excellent high temperature stability of this material is seen up to about 1000°C, above which it slowly loses water. In Figure 3, a curve of muscovite mica is shown. Although it does not possess the same stability as phlogopite, muscovite does remain stable until about 800°C where it, too, begins to evolve water. A curve of a sample of KAPTON, a DuPont polyimide film, is shown in Figure 4. KAPTON starts decomposing at about 525°C, which is in the temperature range of thermal battery operation.

After these initial TG experiments on phlogopite and muscovite mica and on KAPTON and several other possible substitute materials, the TG heating programs were modified to simulate an actual thermal battery temperature vs. time profile as closely as possible. In Figures 5 through 21, the sample was heated at100°C/min to about 500°C and then isothermally for a total of about 30 minutes. At this high heating rate, the furnace would "overshoot" the 500°C limit, thus heating the sample to about 530°C-550°C. This overshoot could have been eliminated by a simple adjustment of the furnace proportional band control. However, the overshoot was considered a good simulation of a thermal battery temperature profile and was therefore retained as part of the heating program. Instead of programming the TGA to simulate the temperature decrease during an actual discharge as the battery slowly cools, the sample was held isothermally at about 500°C; thus, these samples remained at high temperatures longer than they would in an actual battery. In Figures 22 through 28, some samples were rerun by a slightly different method, whereby they were inserted for 30 minutes into a preheated 500°C furnace to achieve a faster rate of sample temperature increase. For samples run by both heating programs, the results were virtually identical although a slightly greater weight loss usually occurred in the program with the overshoot because of the higher temperature achieved and the slightly longer time at the 500°C temperature during isothermal operation. The solid curve is a plot of sample weight remaining vs. time; the dashed curve is a plot of sample temperature vs. time. The time to reach maximum temperature, the temperature during the isothermal heating mode, and the mass remaining after 30 minutes are printed on the curves. The TG results are summarized in Table 2. Discharge results of batteries constructed with several of the insulating materials are summarized in Figures 29 through 37 wherein battery potential and temperature (TC #1) are plotted versus discharge time. All of the batteries, except for the one with the NOMEX 418 insulator, performed comparably to the phlogopite mica battery, to a 24V cutoff, during this short discharge period.

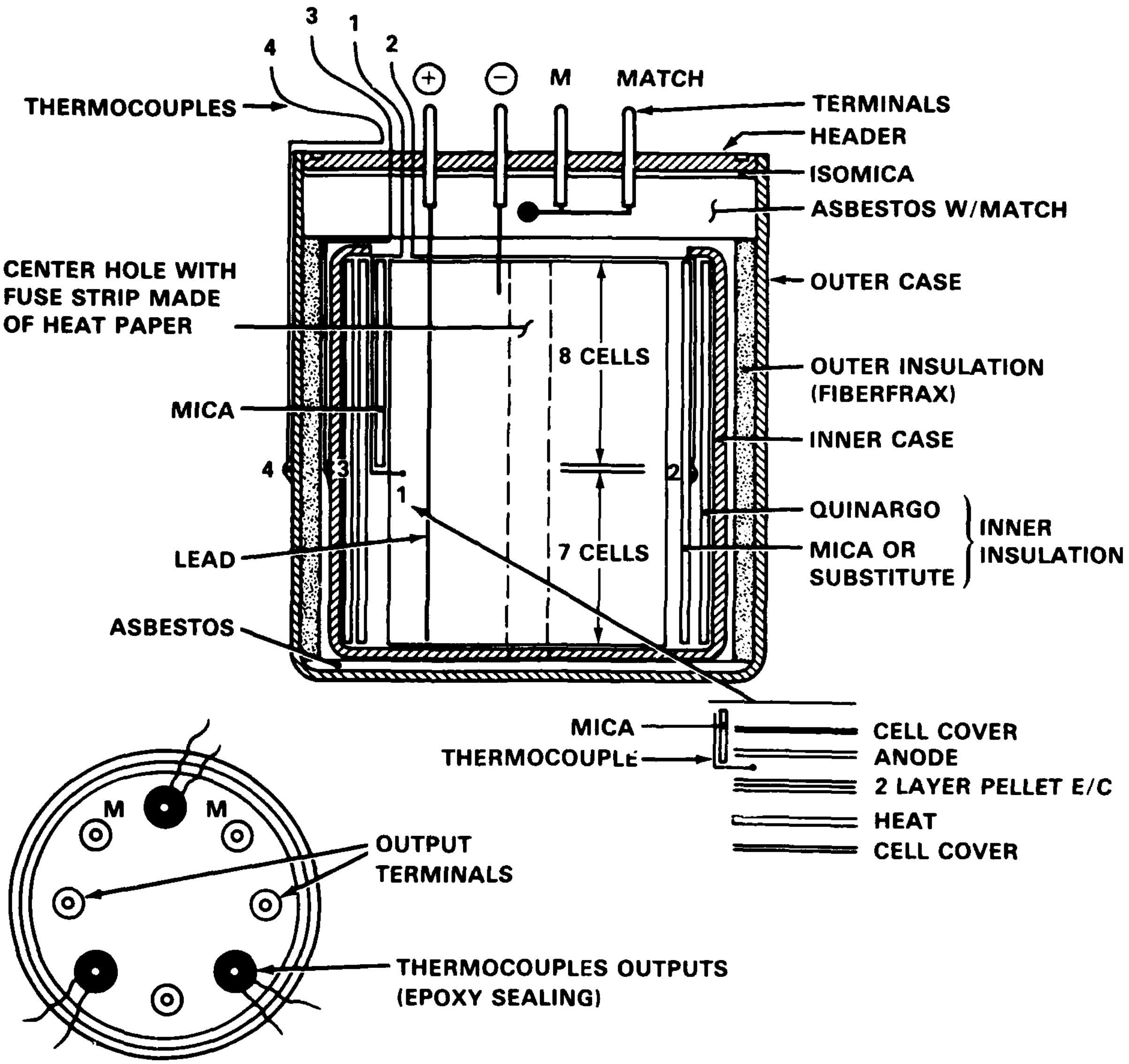
CHAPTER 4

CONCLUSIONS

Of all the materials tested as possible replacements for phlogopite mica as electrical insulators in thermal batteries, only muscovite mica has comparable high temperature stability and electrical properties. Although its decomposition temperature is about 200°C below that of phlogopite mica (800°C vs 1000°C), this is still much higher than the average temperature inside a thermal battery during normal discharge. The discharge behavior of the batteries constructed with muscovite mica insulation was identical to that of the phlogopite mica batteries.

As seen in the TG curves, the other candidate insulators all begin decomposing at or below thermal battery operating temperatures. Post mortem analyses of these insulators from discharged batteries showed various stages of degradation. However, most of these batteries performed as well as the standard phlogopite mica batteries. Most of the degradation is a consequence of the insulators' contact with the high temperature cell stack for a long period after termination of the discharge as the battery slowly cools. Thus, it appears that several of the insulators could be used in short life thermal batteries (2-3 minutes). For longer life thermal batteries (10-30 minutes), actual configurational tests would be required to assess the insulators' capabilities. A summary of the results and recommendations for the eighteen high-temperature insulating materials included in this study is given in Table 3.

We are presently working on alternative methods to achieve electrical insulation of the cell stack in thermal batteries. One possible method is to use a high temperature inorganic polymer coating on the inside of the can; another is to use an anodized aluminum can, instead of mica, as electrical insulation.



NOTES:

1. FIRST THERMOCOUPLE PLACED BETWEEN THE ANODE AND THE TWO LAYER PELLET OF THE 9TH CELL (FROM TOP OF STACK).
2. SECOND THERMOCOUPLE IS BETWEEN THE MICA AND QUINARGO INSULATION OF THE INNER CASE.
3. THIRD THERMOCOUPLE IS BETWEEN THE FIBERFRAX INSULATION OF THE OUTER CASE AND THE OUTSIDE OF THE INNER CASE.
4. THE FOURTH THERMOCOUPLE IS ON THE OUTSIDE OF THE OUTER CASE.
5. ALL THERMOCOUPLES ARE APPROXIMATELY AT THE SAME LEVEL. (APPROX. MIDWAY DOWN THE STACK)

FIGURE 1. CONSTRUCTION OF TEST BATTERIES SHOWING POSITION OF THERMOCOUPLES

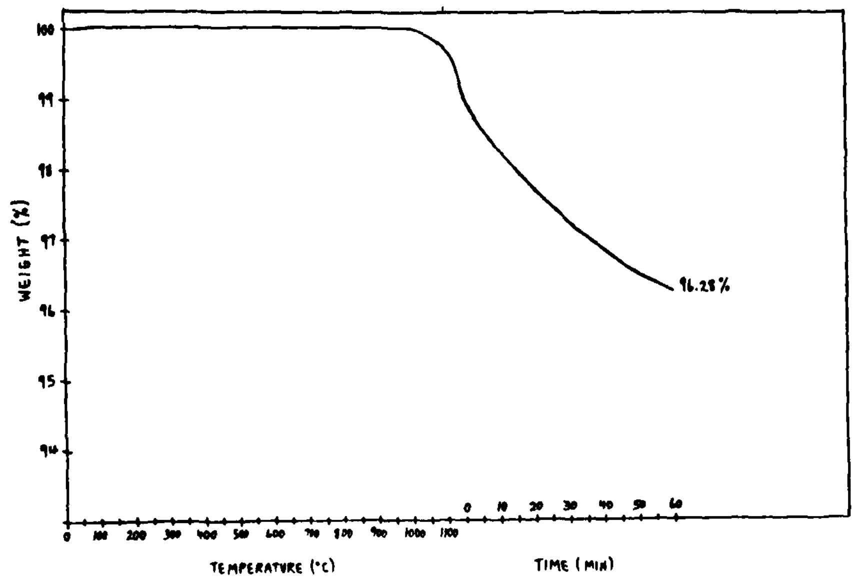


FIGURE 2. TG CURVE OF PHLOGOPITE MICA, 80.31 MG, 100° C/MIN (20°-900°C), 5° C/MIN (900°-1150°C), ISOTHERMAL (1150°C), 50 CC/MIN Ar

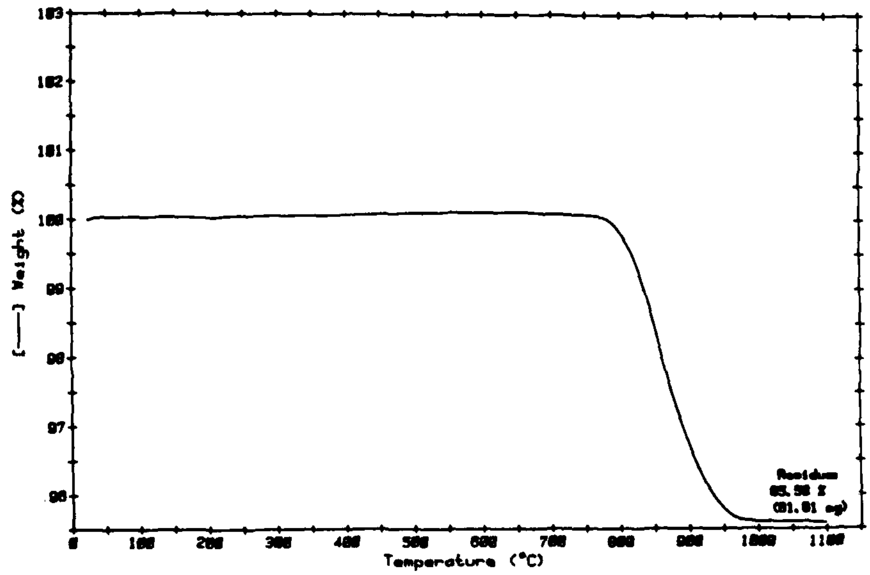


FIGURE 3. TG CURVE OF MUSCOVITE MICA, 84.77 MG, 20° C/MIN, 50 CC/MIN Ar

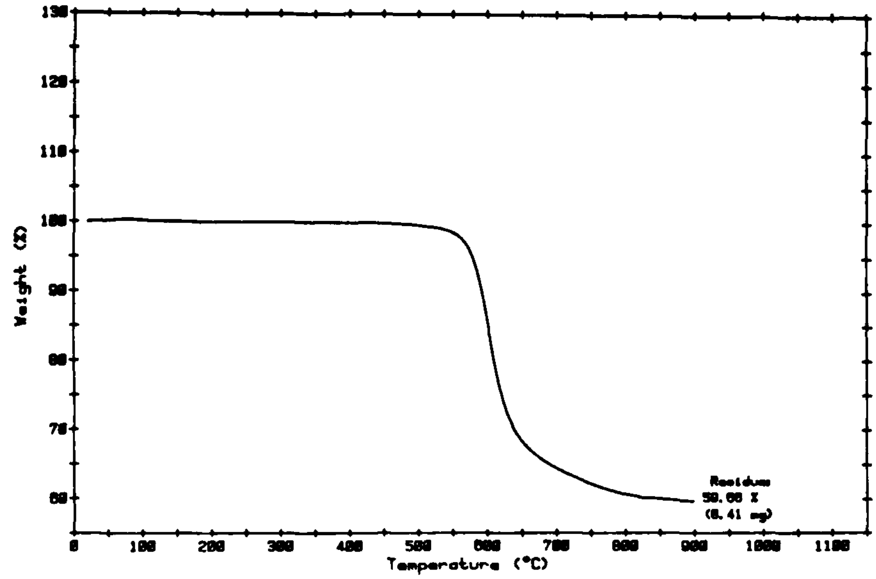


FIGURE 4. TG CURVE OF KAPTON 300 H, 14.10 MG, 20° C/MIN, 50 CC/MIN Ar

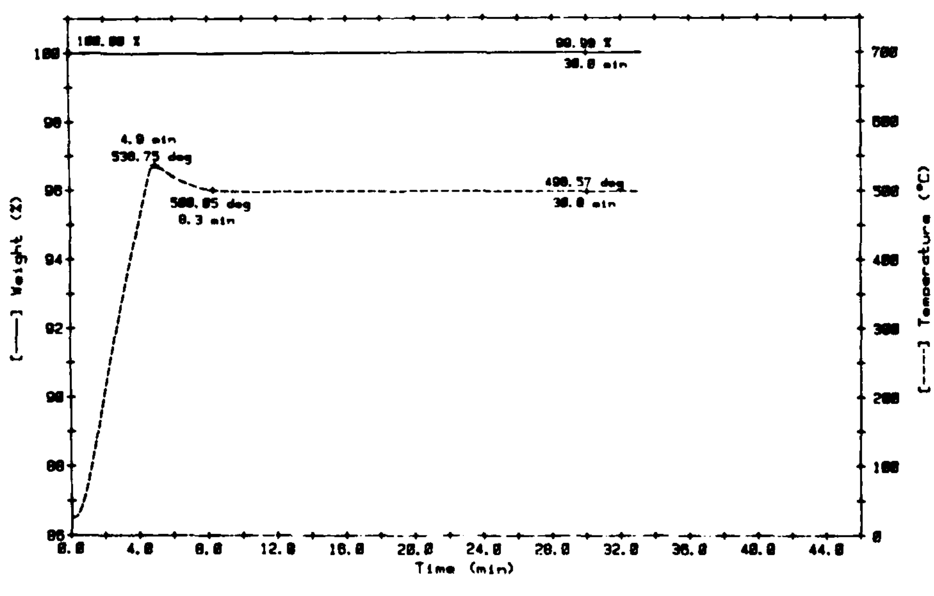


FIGURE 5. TG CURVE OF PHLOGOPITE MICA, 80.70 MG, ISOTHERMAL (500°C), 50 CC/MIN He

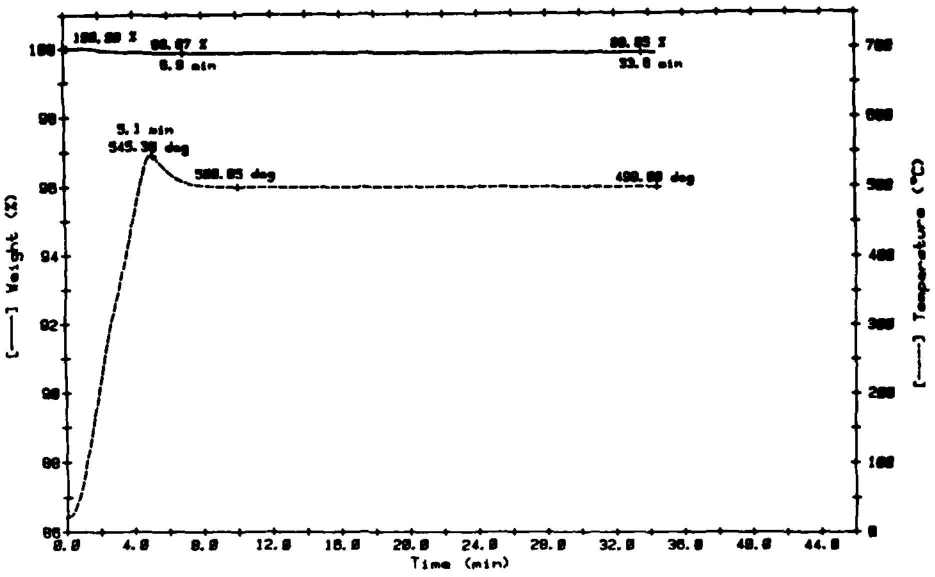


FIGURE 6. TG CURVE OF MUSCOVITE MICA, 68.07 MG, ISOTHERMAL (500°C), 50 CC/MIN He

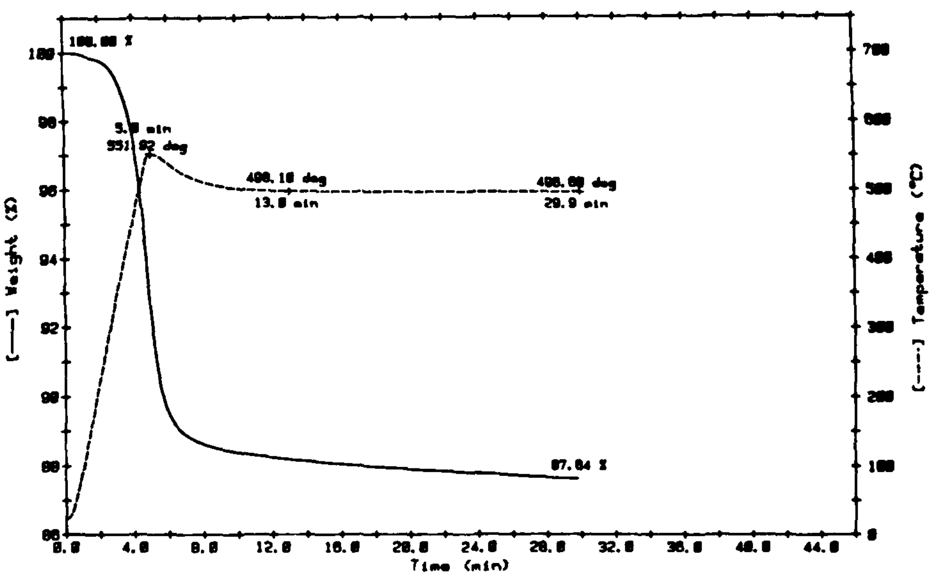


FIGURE 7. TG CURVE OF ESSEX P/N 11827, 77.15 MG, ISOTHERMAL (500°C), 50 CC/MIN He

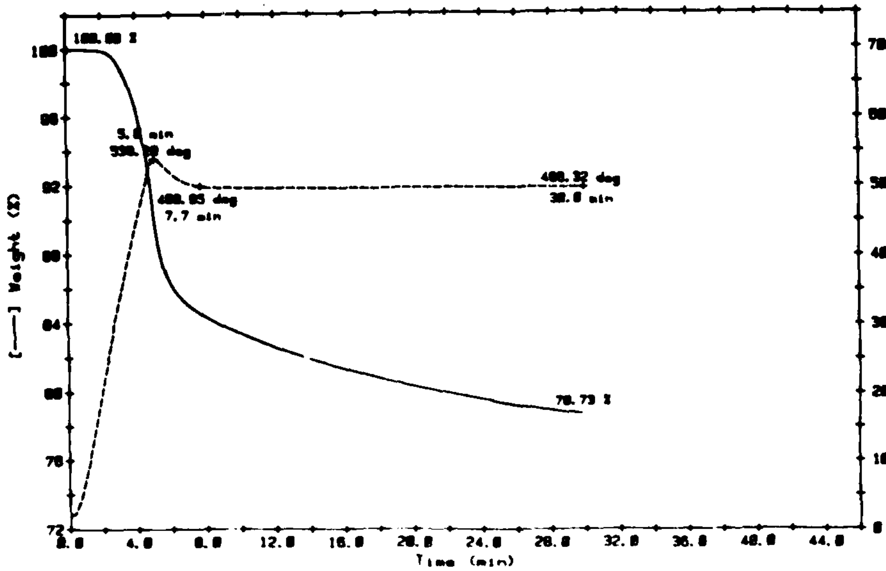
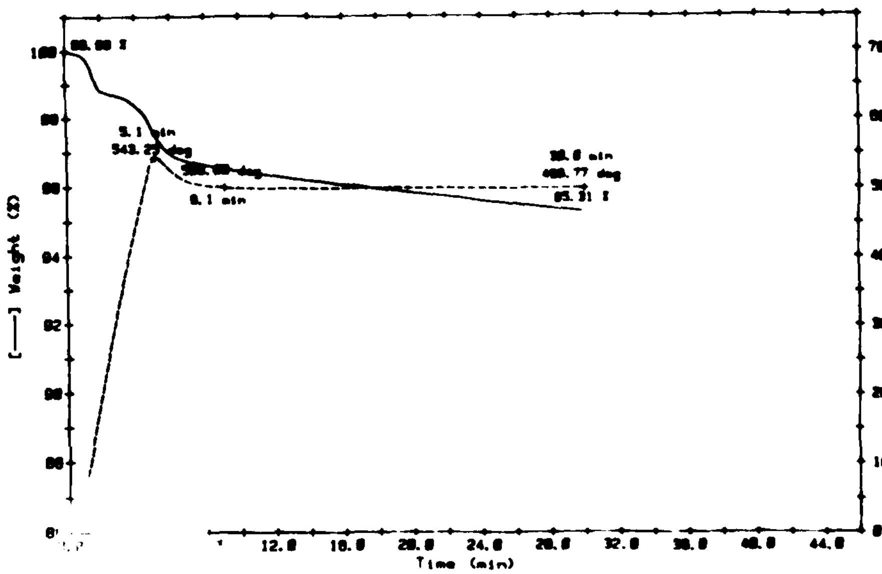


FIGURE 8. TG CURVE OF ESSEX P/N 11064, 75.62 MG, ISOTHERMAL (500°C), 50 CC/MIN He



FIGU[illegible] [illegible] CURVE OF KAPTON 300 H, 19.98 MG, ISOTHERMAL [illegible]500°C), 50 CC/MIN He

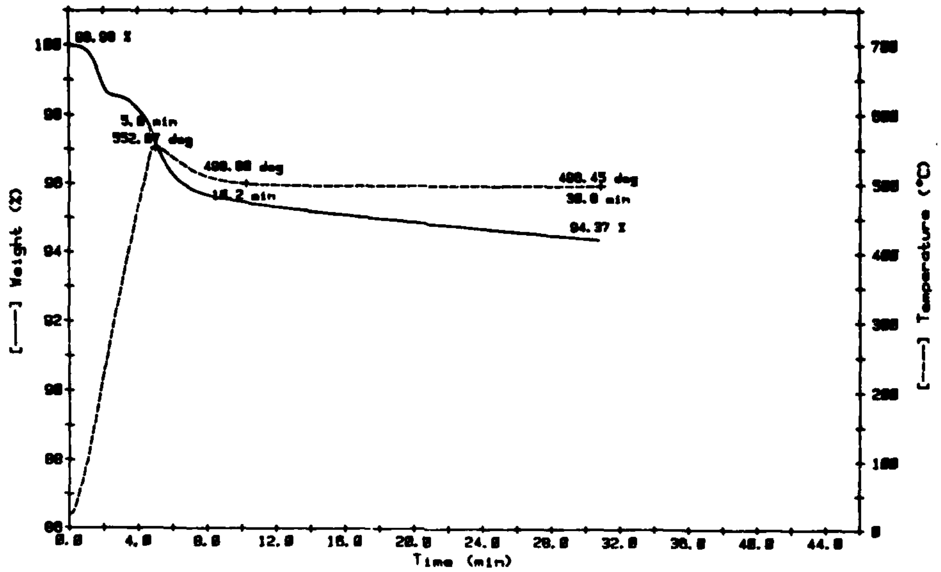


FIGURE 10. TG CURVE OF KAPTON 500 H, 52.40 MG, ISOTHERMAL (500°C), 50 CC/MIN He

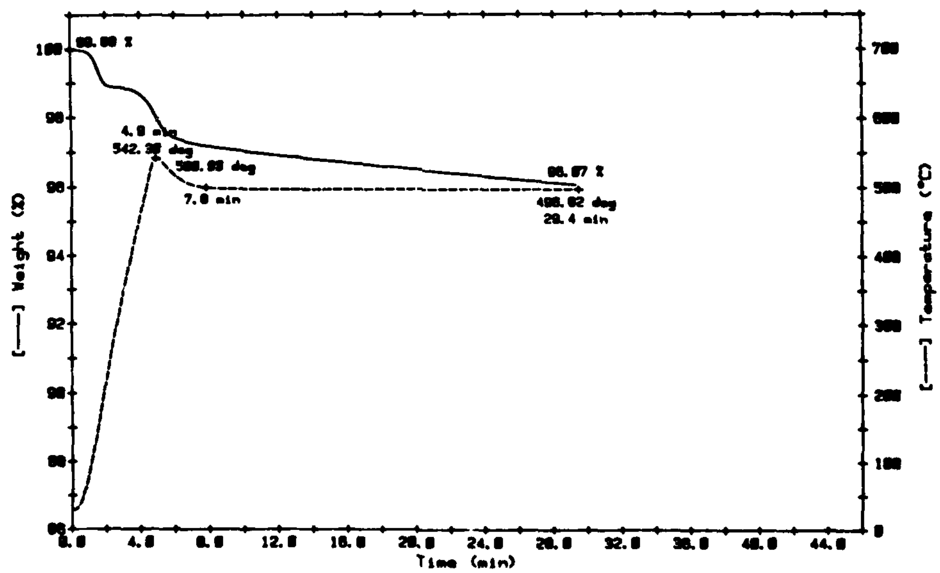


FIGURE 11. TG CURVE OF KAPTON 300 V, 33.33 MG, ISOTHERMAL (500°C), 50 CC/MIN He

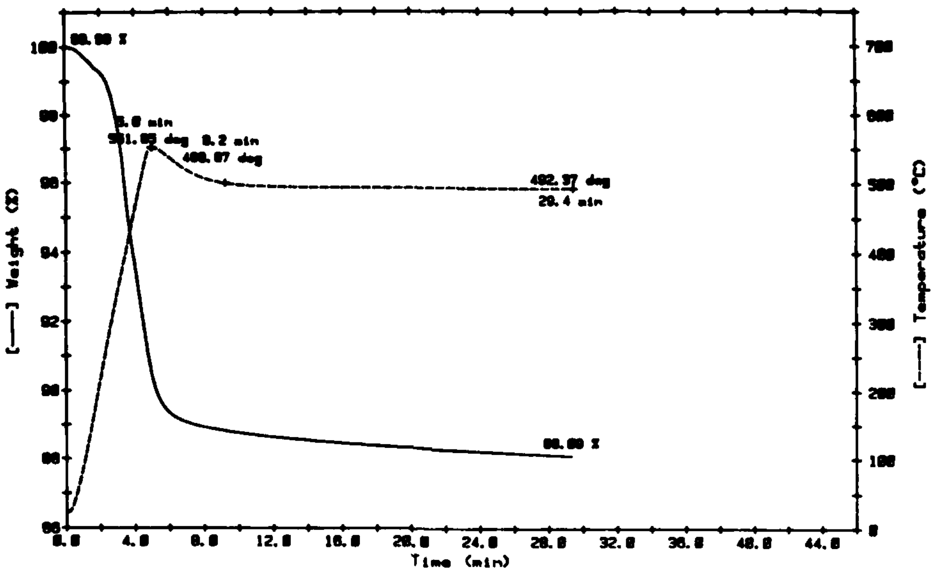


FIGURE 12. TG CURVE OF QUINTERRA T3, 40.44 MG, ISOTHERMAL (500°C), 50 CC/MIN He

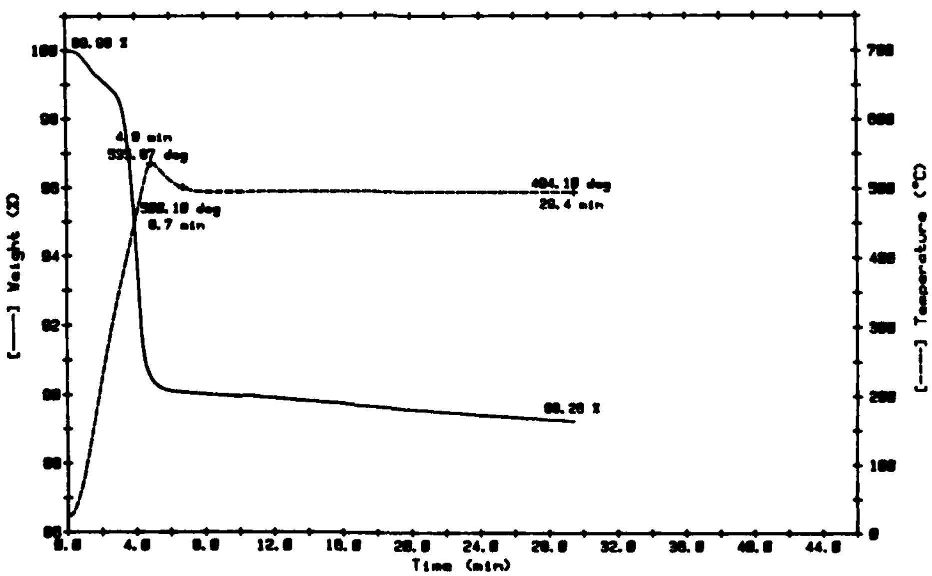


FIGURE 13. TG CURVE OF QUINORGO 5000, 25.68 MG, ISOTHERMAL (500°C), 50 CC/MIN He

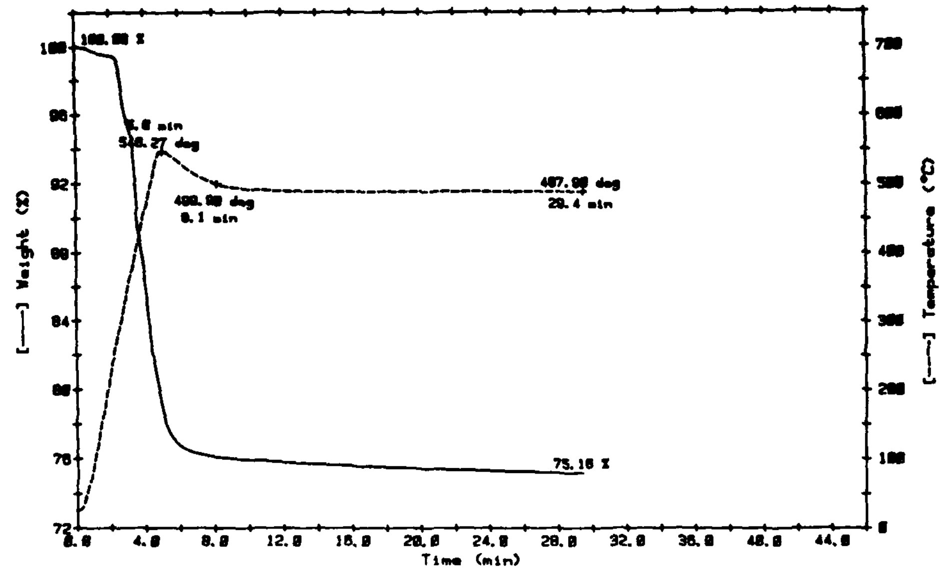


FIG. 14

FIGURE 14. TG CURVE OF CE QUIN 1, 23.01 MG, ISOTHERMAL (500°C), 50 CC/MIN He

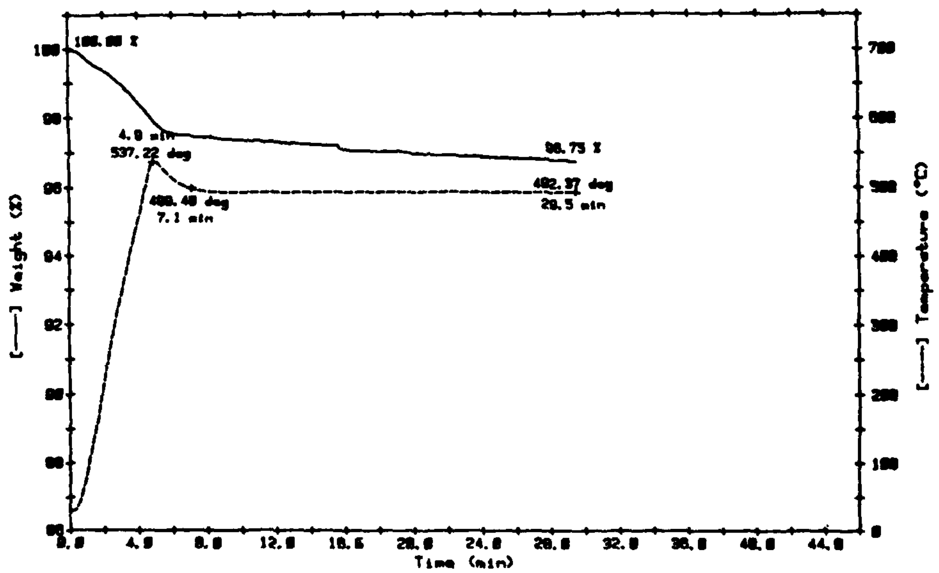


FIGURE 15. TG CURVE OF QUINT-T, 8.61 MG, ISOTHERMAL (500°C), 50 CC/MIN He

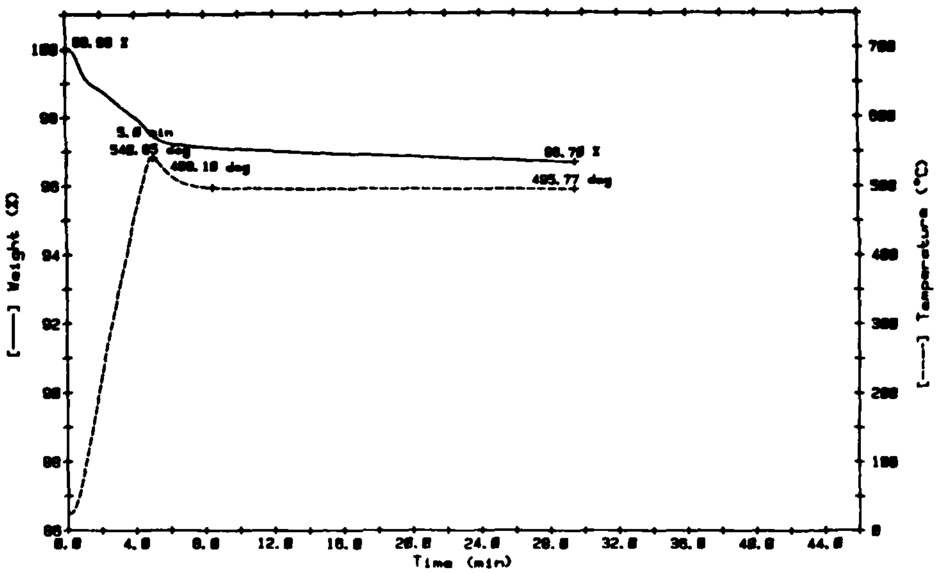


FIGURE 16. TG CURVE OF FUEL CELL PAPER, 15.16 MG, ISOTHERMAL (500°C), 50 CC/MIN He

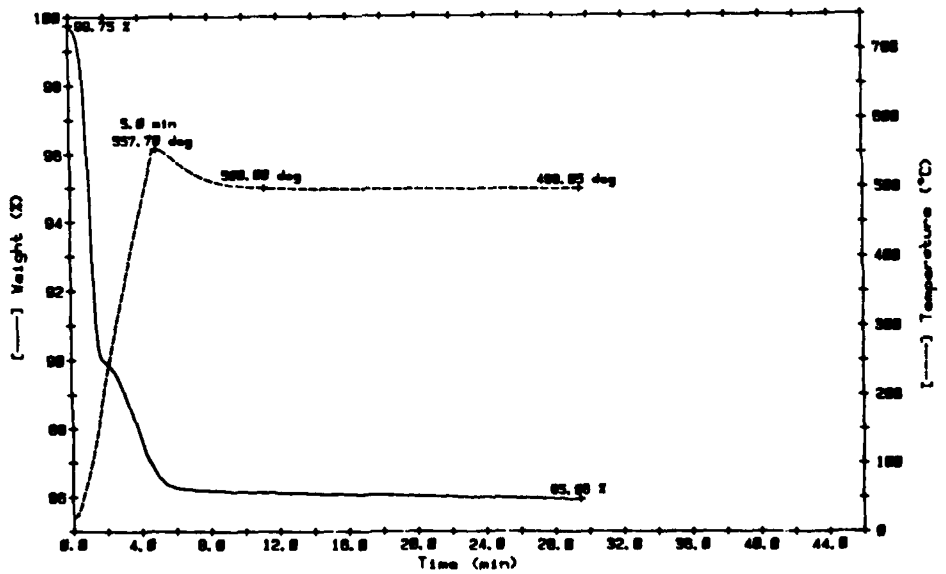


FIGURE 17. TG CURVE OF HAVEG SILTEMP TAPE, 12.23 MG, ISOTHERMAL (500°C), 50 CC/MIN He

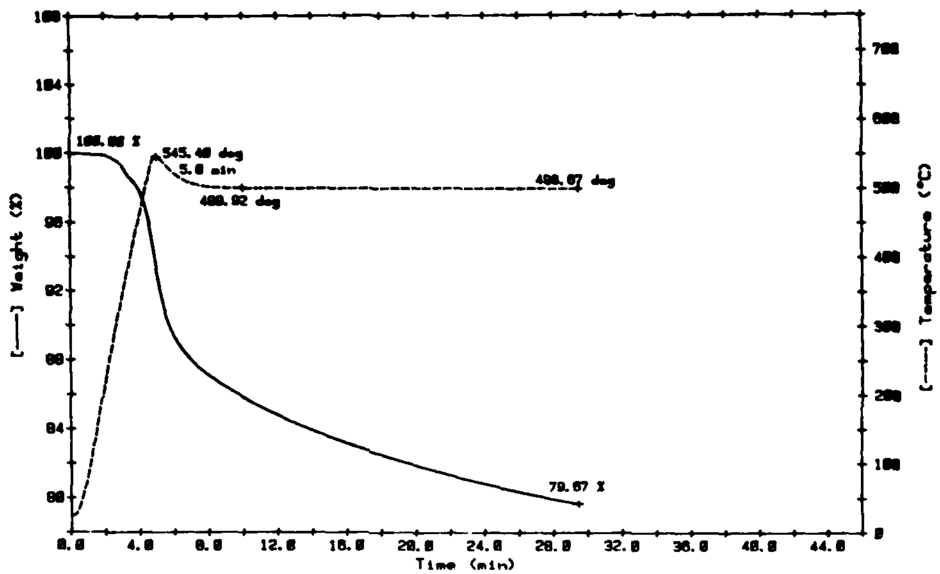


FIGURE 18. TG CURVE OF SILICONE & VITON #1606, 44.16 MG, ISOTHERMAL (500°C), 50 CC/MIN He

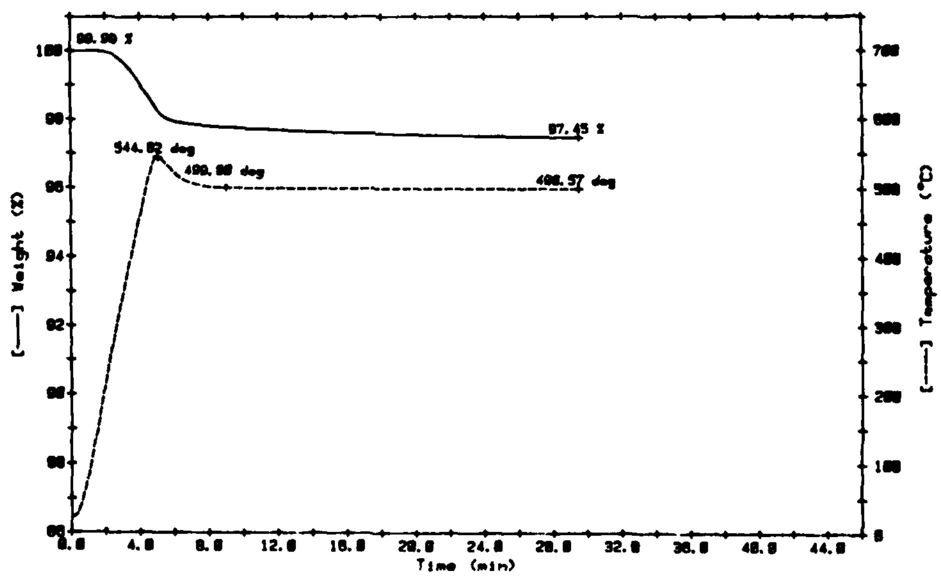


FIGURE 19. TG CURVE OF ESSEX P/N 470005, 53.69 MG, ISOTHERMAL (500°C), 50 CC/MIN He

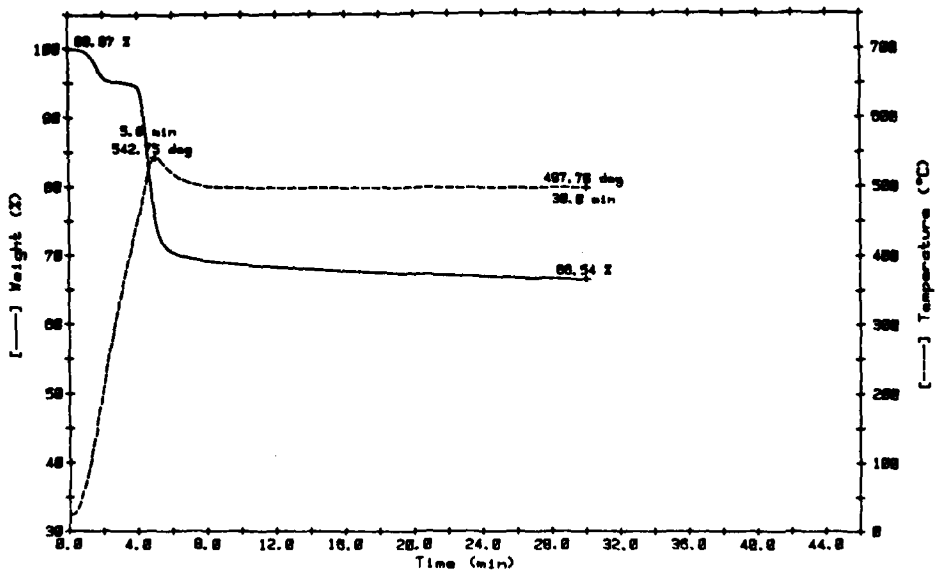


FIGURE 20. TG CURVE OF NOMEX 410, 40.33 MG, ISOTHERMAL (500°C), 50 CC/MIN He

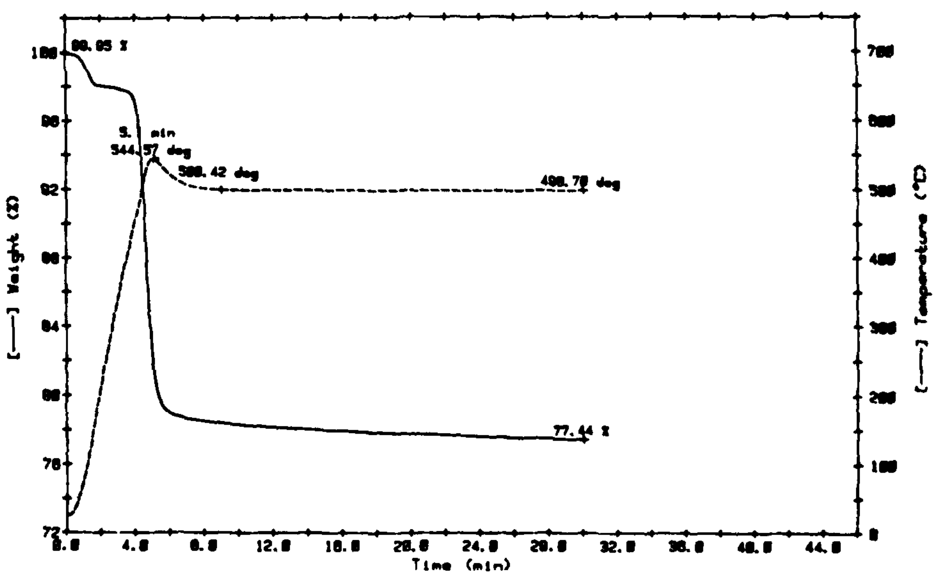


FIGURE 21. TG CURVE OF NOMEX 418, 46.24 MG, ISOTHERMAL (500°C), 50 CC/MIN He

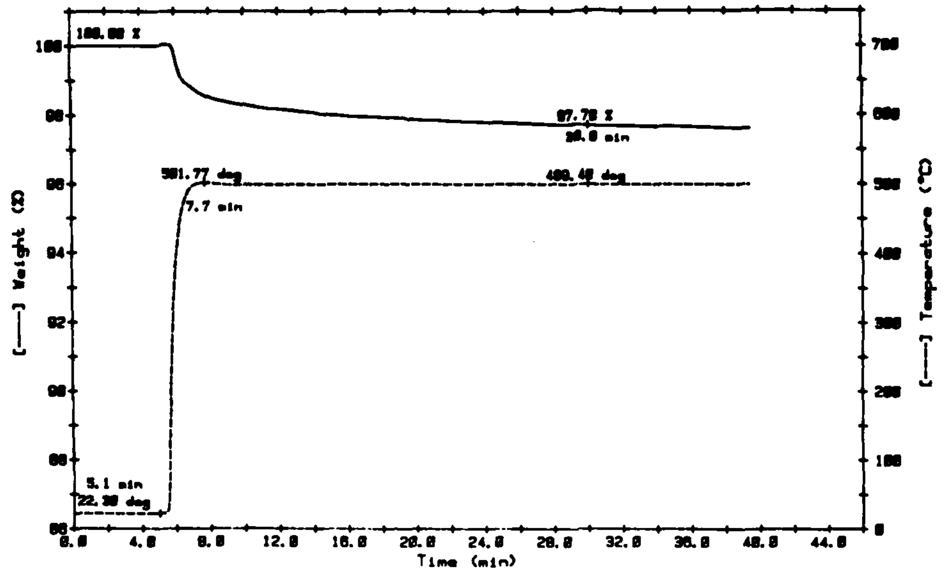


FIGURE 22. TG CURVE OF ESSEX P/N 470005, 41.92 MG, ISOTHERMAL (500°C), 50 CC/MIN He

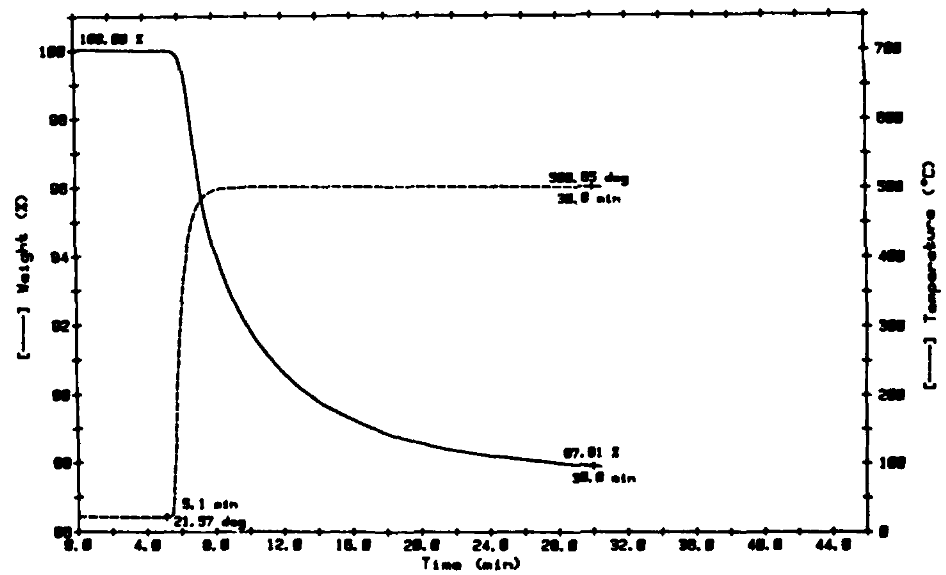


FIGURE 23. TG CURVE OF ESSEX P/N 11827, 43.92 MG, ISOTHERMAL (500°C), 50 CC/MIN He

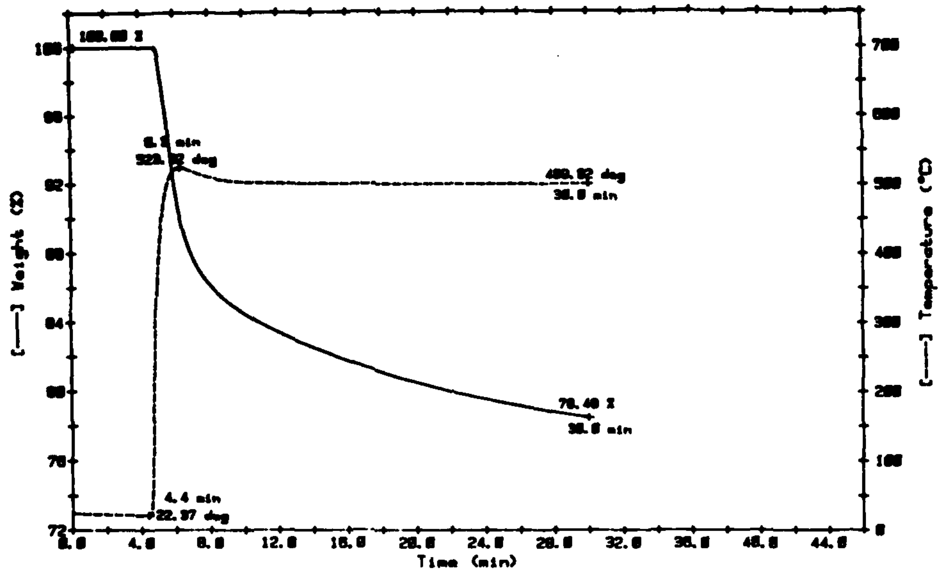


FIGURE 24. TG CURVE OF ESSEX P/N 11054, 48.38 MG, ISOTHERMAL (500°C), 50 CC/MIN He

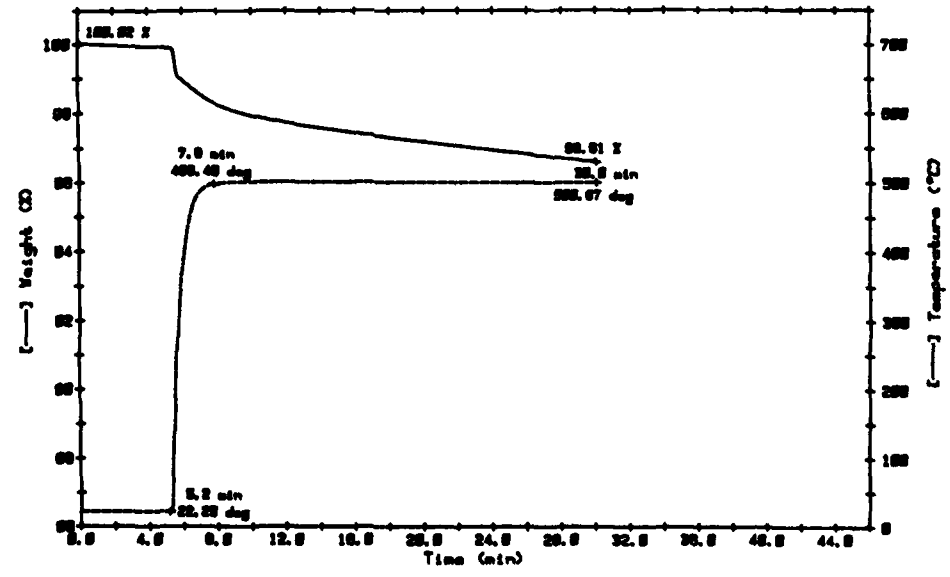


FIGURE 25. TG CURVE OF KAPTON 300 H, 32.46 MG, ISOTHERMAL (500°C), 50 CC/MIN He

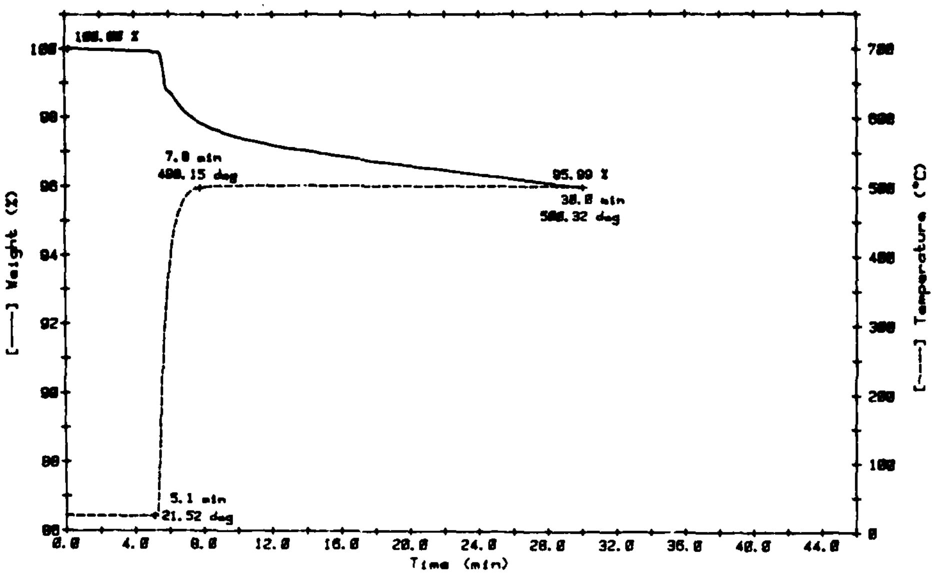


FIGURE 26. TG CURVE OF KAPTON 500 H, 45.35 MG, ISOTHERMAL (500°C), 50 CC/MIN He

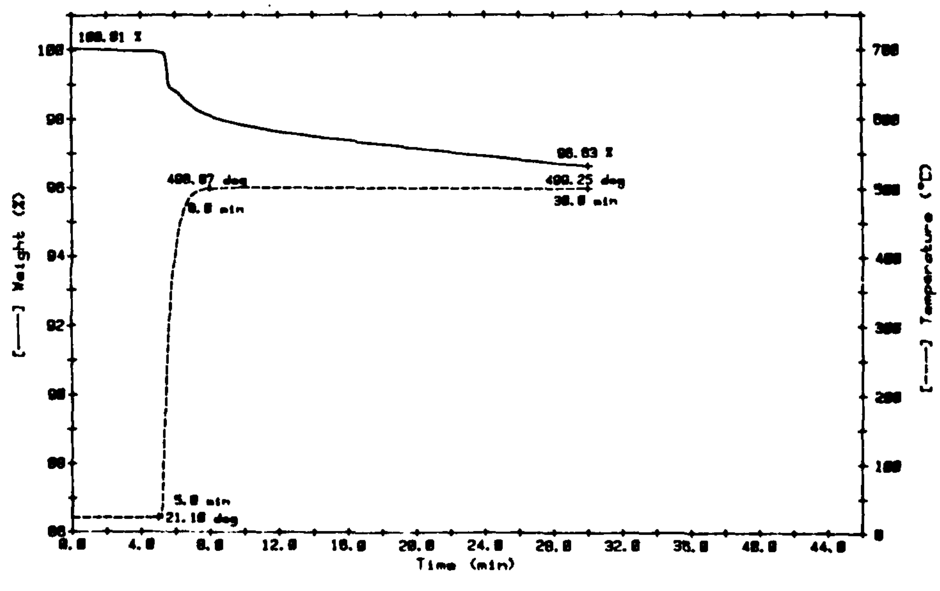


FIGURE 27. TG CURVE OF KAPTON 300 V, 36.95 MG, ISOTHERMAL (500°C), 50 CC/MIN He

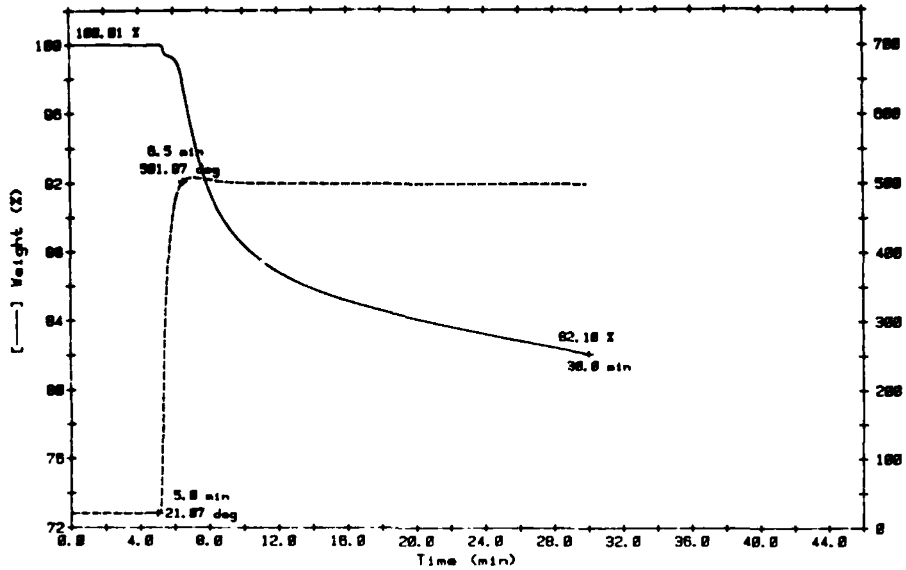


FIGURE 28. TG CURVE OF KAPTON 300 F, 30.30 MG, ISOTHERMAL (500°C), 50 CC/MIN He

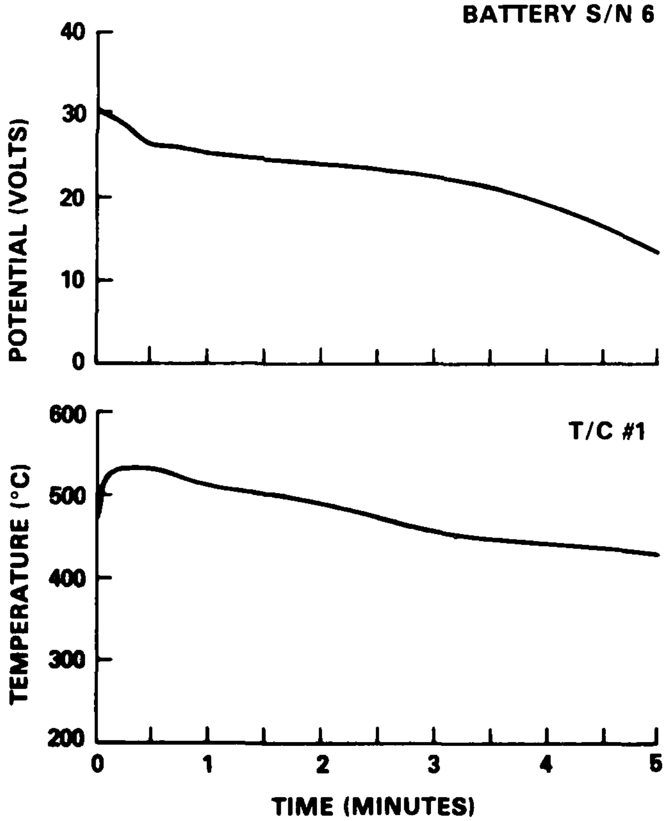


FIGURE 29. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH PHLOGOPITE MICA INSULATOR

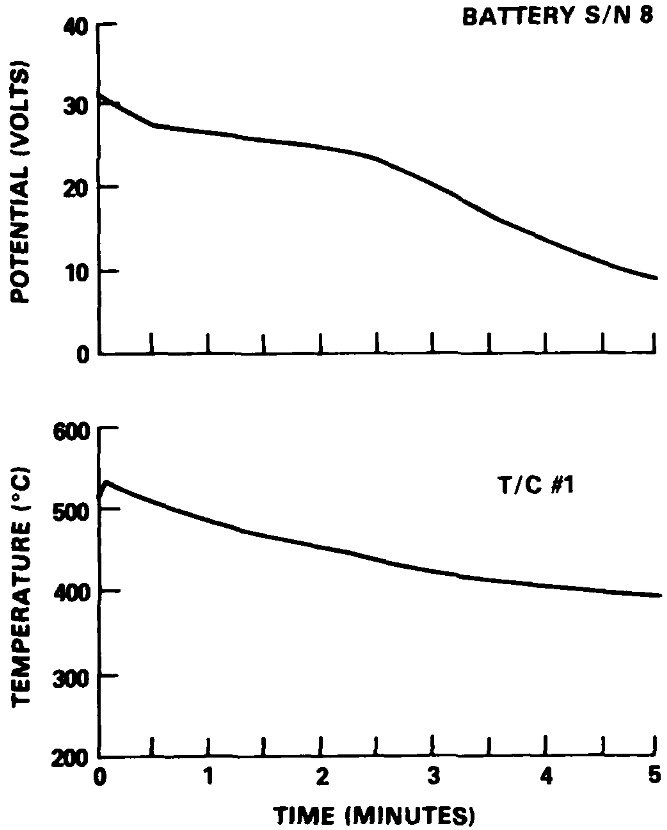


FIGURE 30. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH KAPTON 300V INSULATOR

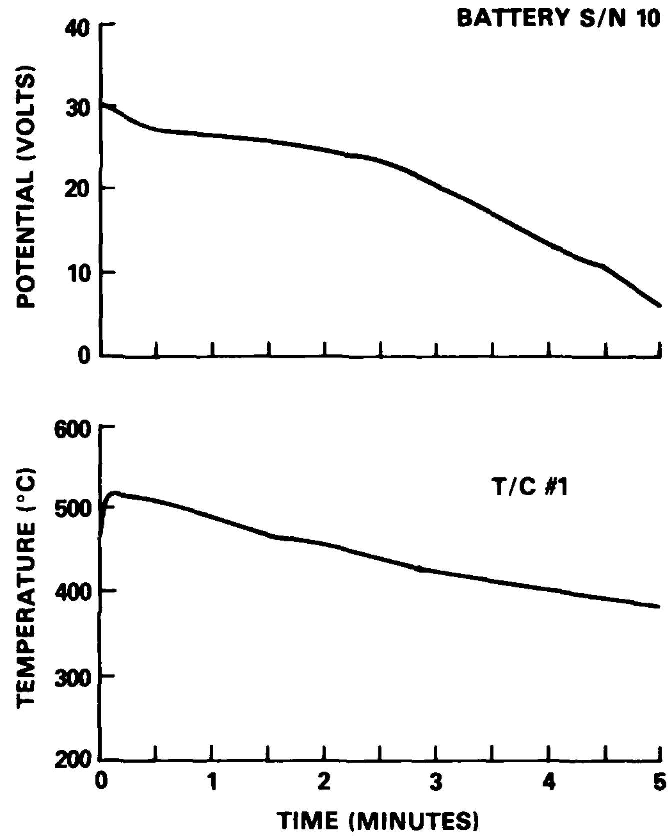


FIGURE 31. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH KAPTON 500H INSULATOR

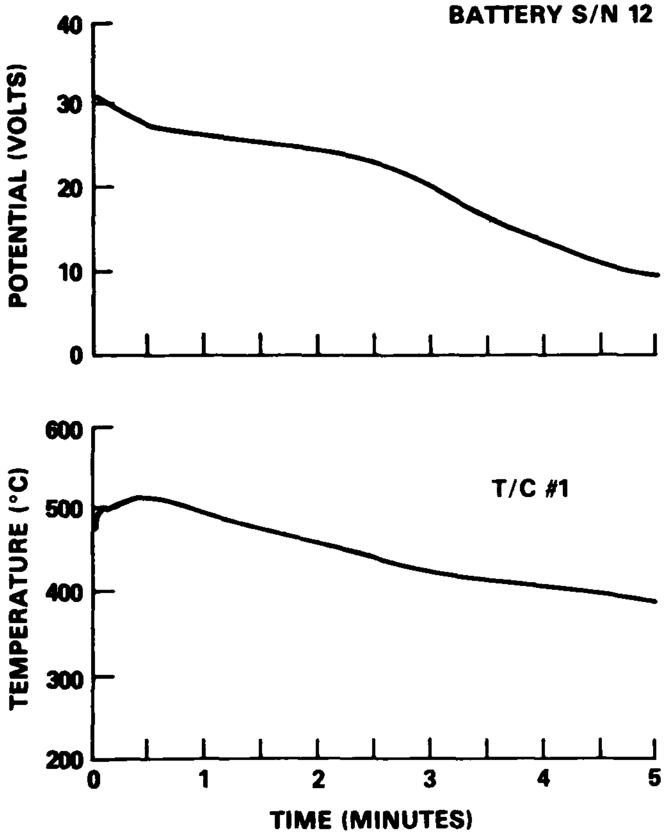


FIGURE 32. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH KAPTON 300H INSULATOR

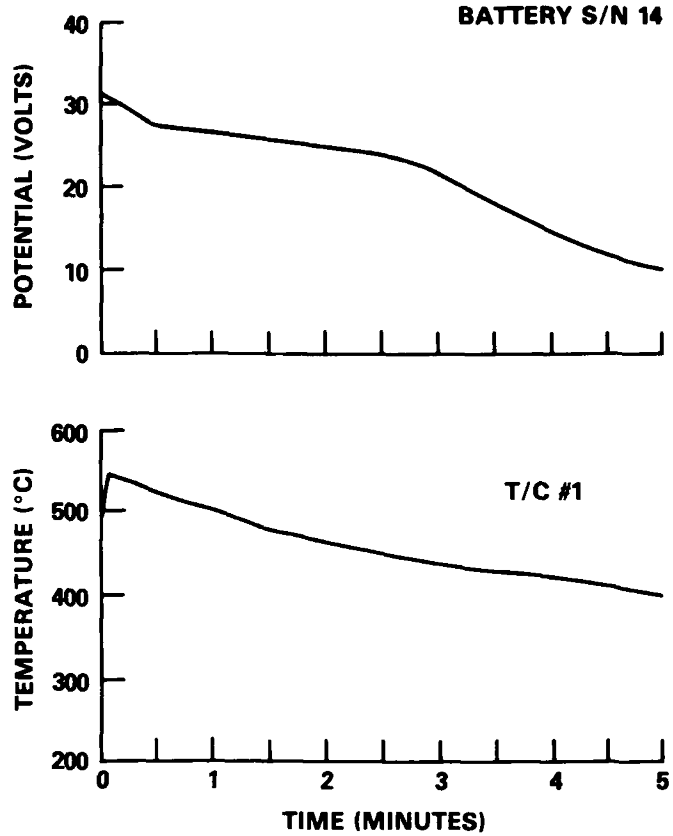


FIGURE 33. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH MUSCOVITE MICA INSULATOR

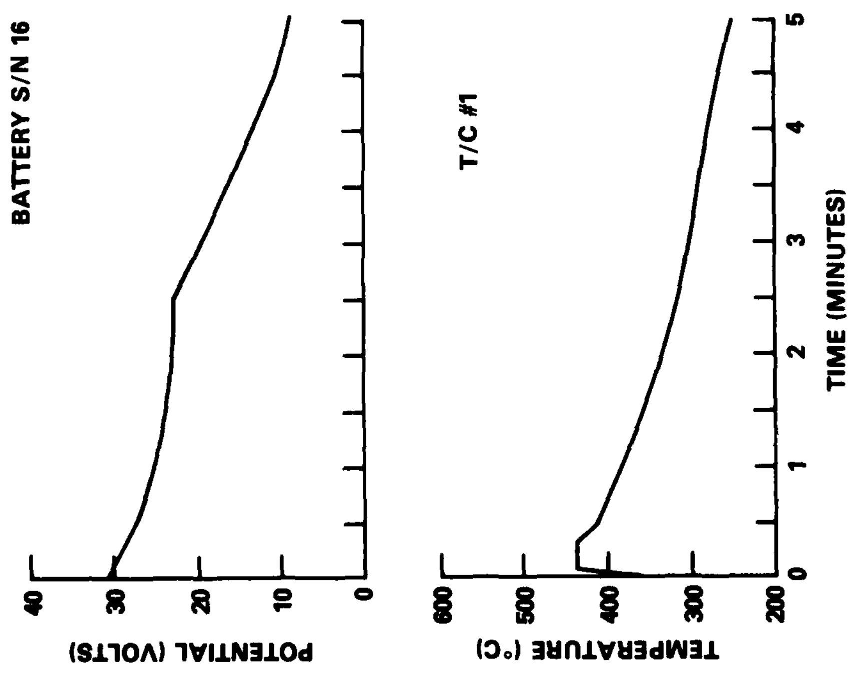


FIGURE 34. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH ESSEX P/N 11827 INSULATOR

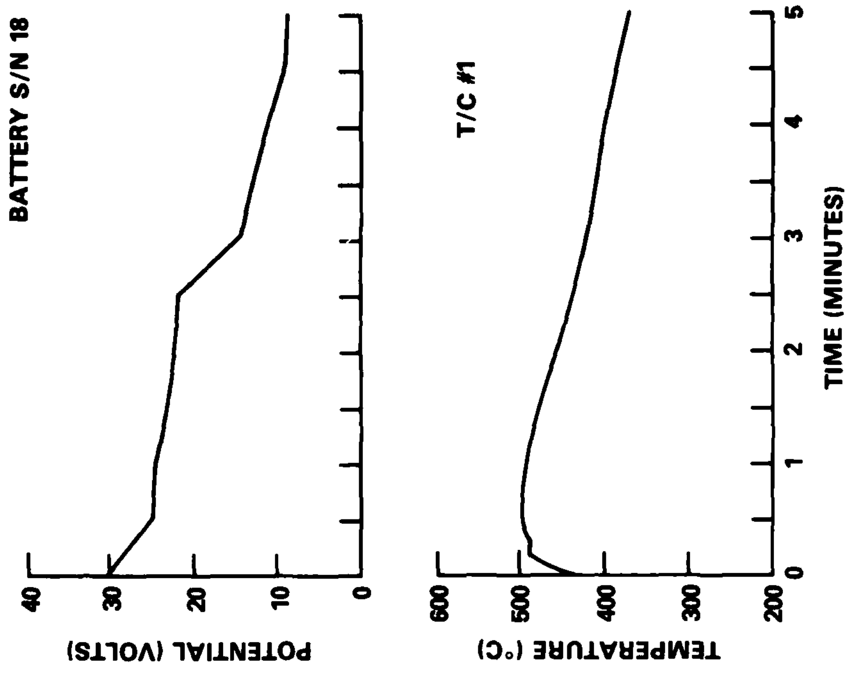


FIGURE 35. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH ESSEX P/N 470005 INSULATOR

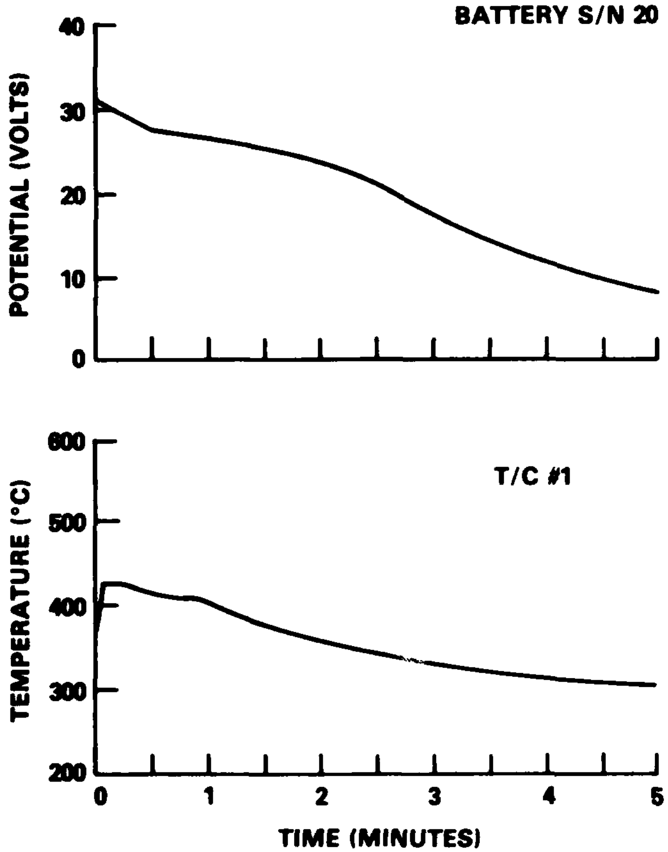


FIGURE 36. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH ESSEX P/N 11064 INSULATOR

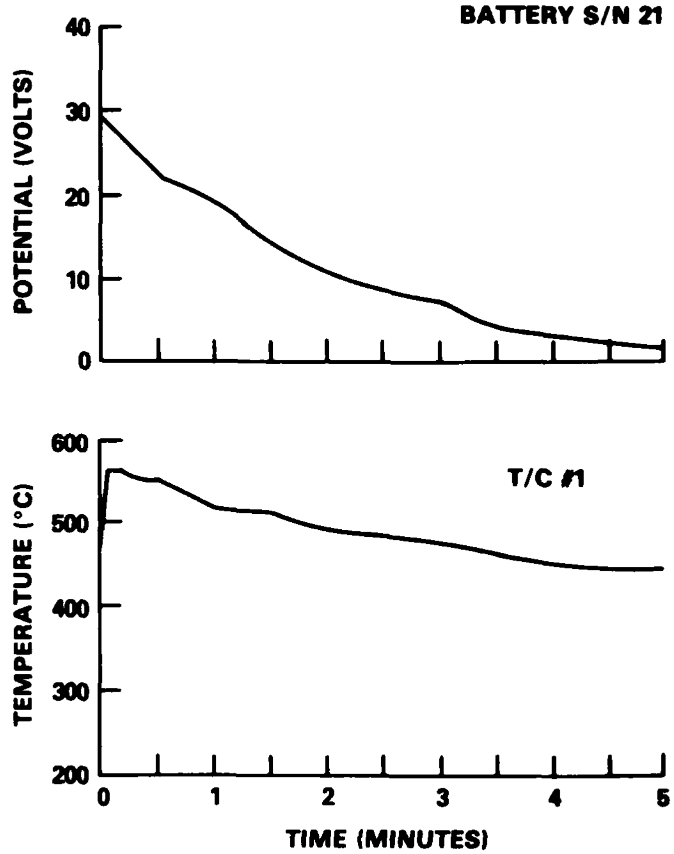


FIGURE 37. POTENTIAL AND TEMPERATURE DURING DISCHARGE OF BATTERY WITH NOMEX 418 INSULATOR

TABLE 1. COMPOSITION OF TEST MATERIALS

SAMPLE	COMPOSITION	COMPANY
PHLOGOPITE MICA	A COMPLEX HYDROUS ALUMINUM SILICATE	
MUSCOVITE MICA	A COMPLEX HYDROUS ALUMINUM SILICATE	
KAPTON 300H	POLYIMIDE FILM	DUPONT COMPANY
KAPTON 500H	POLYIMIDE FILM	DUPONT COMPANY
KAPTON 300V	POLYIMIDE FILM	DUPONT COMPANY
KAPTON 300F	POLYIMIDE FILM COATED WITH TEFLON	DUPONT COMPANY
ESSEX P/N 11827	KAPTON POLYIMIDE-MICA-GLASS CLOTH COMPOSITE	UNITED TECHNOLOGIES ESSEX GROUP
ESSEX P/N 11054	SILICONE-MICA-GLASS CLOTH	UNITED TECHNOLOGIES ESSEX GROUP
ESSEX P/N 470005	ARAMID POLYMER + PLATELET MICA	UNITED TECHNOLOGIES ESSEX GROUP
NOMEX 410	ARAMID POLYMER + PLATELET MICA	UNITED TECHNOLOGIES ESSEX GROUP
NOMEX 418	ARAMID POLYMER + PLATELET MICA	UNITED TECHNOLOGIES ESSEX GROUP
QUINTERRA T3	MAGNESIUM SILICATE FIBER (ASBESTOS FIBER PAPER)	QUIN-T CORPORATION
QUINORGO 5000	MAGNESIUM SILICATE FIBER (ASBESTOS FIBER + ELASTOMERIC BINDER)	QUIN-T CORPORATION
CE QUIN I	ALUMINUM SILICATE (NON-ASBESTOS)	QUIN-T CORPORATION
FUEL CELL PAPER	MAGNESIUM SILICATE FIBER (ASBESTOS FIBER)	QUIN-T CORPORATION
QUIN-T		QUIN-T CORPORATION
CHR SILICONE VITON 1601		CHR INDUSTRIES, INC.
SILTEMP TAPE		HAVEG CORPORATION

TABLE 2. THERMOGRAVIMETRY RESULTS

INSULATOR	THICKNESS (mils)	TG* WEIGHT LOSS	TG** % WEIGHT LOSS
PHLOGOPITE MICA	4	0.01	
MUSCOVITE MICA	4	0.15	
KAPTON 300 V	3	3.93	3.37
KAPTON 300 H	3	4.69	3.39
KAPTON 500 H	5	5.63	4.01
KAPTON 300 F	3		17.90
ESSEX P/N 11827	9	12.36	12.09
ESSEX P/N 11054	10	21.27	21.46
ESSEX 470005	5	2.55	2.30
NOMEX 410	5	22.56	
NOMEX 418	10	33.46	
CHR SILICONE VITON 1606	6	20.33	
SILTEMP TAPE	20	14.04	
QUINTERRA T3	4	11.91	
QUINORGO 5000	5	10.74	
Ce QUIN I	5	24.84	
QUIN-T		3.25	
FUEL CELL PAPER	10	3.30	

*Figures 5 through 21
**Figures 22 through 28

TABLE 3. SUMMARY OF RESULTS AND RECOMMENDATIONS

INSULATOR	THICKNESS (mils)	SUITABILITY FOR THERMAL BATTERY USE
PHLOGOPITE MICA	4	EXCELLENT THERMAL STABILITY. SUITABLE FOR USE TO 1000°C.
MUSCOVITE MICA		GOOD THERMAL STABILITY RECOMMENDED FOR USE TO 800°C
KAPTON 300 V	3	PARTIAL DECOMPOSITION AT 500°C. SOME SHRINKAGE AND DEFORMATION IN BATTERY TESTS. RECOMMENDED ONLY WHEN BATTERY DISCHARGE LIFE IS LESS THAN THREE MINUTES.
KAPTON 300 H	3	
KAPTON 500 H	5	
KAPTON 300 F	3	TEFLON COATING REACTS WITH LITHIUM ANODE. NOT RECOMMENDED.
ESSEX P/N 11827	9	EXCESSIVE THERMAL DECOMPOSITION AT 500°C RESULTING IN POOR ELECTRICAL INSULATION PROPERTIES. NOT RECOMMENDED.
ESSEX P/N 11054	10	
ESSEX P/N 470005		
NOMEX 410	5	
NOMEX 418	10	
CHR SILICONE VITON 1606	6	
SILTEMP TAPE	20	
QUINTERRA T3	4	WETTED BY MOLTEN SALT ELECTROLYTE RESULTING IN POOR ELECTRICAL INSULATION PROPERTIES. NOT RECOMMENDED.
QUINORGO 5000	5	
Ce QUIN I	5	
QUINT-T		
FUEL CELL PAPER	10	

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